Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) is used for structure determination and identification of organic and inorganic compounds. It is particularly useful for determining the presence of functional groups in a molecule.

Principle of Technique

The FTIR spectrometer consists of a broad-band infrared source, an interferometer, and a detector. Light from the source is directed into the interferometer, where a beam splitter transmits 50% of the light to a fixed mirror and reflects 50% to a moving mirror. The light then recombines at the beam splitter, is directed through the sample, and impinges on the detector. The resulting signal is output to a computer for processing. The frequencies of the light absorbed are characteristic of the vibrational modes of the molecules present in the sample, and these in turn depend on the functional groups, the molecular structure, and its conformation. The FTIR instrument differs from a dispersive instrument because it has an interferometer rather than a grating. Because the detector measures only total light, the value at any time (corresponding to different mirror positions) is the sum of the energy from all wavelengths. This interferogram consists of a power vs time spectrum. A Fourier transform maps from the time domain into the wavelength domain. The result is a plot of frequency vs transmittance or absorbance. From this plot, molecular structure can be determined and, in many cases, a positive identification of the particular compound can be made.

Samples

Form. Almost any solid, liquid, or gas can be analyzed.

Size. Solid samples must be at least 10 mg (less if soluble in an organic solvent) or 1 cm^2 in area for flat surfaces. Liquid samples must be at least 50 µL if neat (less if soluble in an organic solvent). Gas samples must be at least 10 cc in volume.

Preparation. Minimal or no preparation is needed for qualitative analysis. For quantitative analysis, standards need to be mixed and analyzed.

Limitations

Infrared spectroscopy is not usually recommended for mixtures of compounds, although it can be used for the analysis of well-characterized samples with less than 10 components. A compound must be in a matrix that is transparent in the spectral region of interest. The analyte must have a permanent dipole moment in order to be detected.

Aqueous samples are difficult because the absorptivity of the O–H vibration in the mid-infrared region often obscures important analyte absorbances. Water also dissolves most windows used for sample support.

Compounds can be determined at concentrations of a few percent and, with special techniques, at concentrations of less than 0.1%.

Estimated Analysis Time

About 1 to 2 h is normally required to prepare the sample, obtain a spectrum, and interpret the results. Quantitative determination requires additional time to prepare and analyze standards.

Examples of Applications

- Identification of resins, polymers, rubbers, explosives, mock explosives, and surface coatings.
- Identification of contaminants such as plastics, grease, or pump oil.
- Identification of solids (not ideally applicable to complex mixtures).
- Quantitative determination of residual hydrocarbons in Freon flushings of welded vessels.

Capabilities of Related Techniques

Raman spectroscopy may yield similar qualitative and quantitative information, and is applicable to aqueous solutions.

Near-infrared (NIR) spectroscopy typically employs different instrumentation than FTIR. It can be used to measure water and hydroxyl-containing compounds in organic solvents.

Nuclear magnetic resonance spectroscopy may yield more detailed structural information and is applicable to different types of compounds.

Other FTIR Analysis Methods

Infrared Microscopy

Infrared microscopy can be applied to small samples (to 20 microns) in solid matrices with transparent, inhomogeneous portions to identify fibers, coatings, particles, and contaminants on metallic and dielectric substrates (e.g., the composition of each layer of a laminated polymer).

Attenuated Total Reflectance

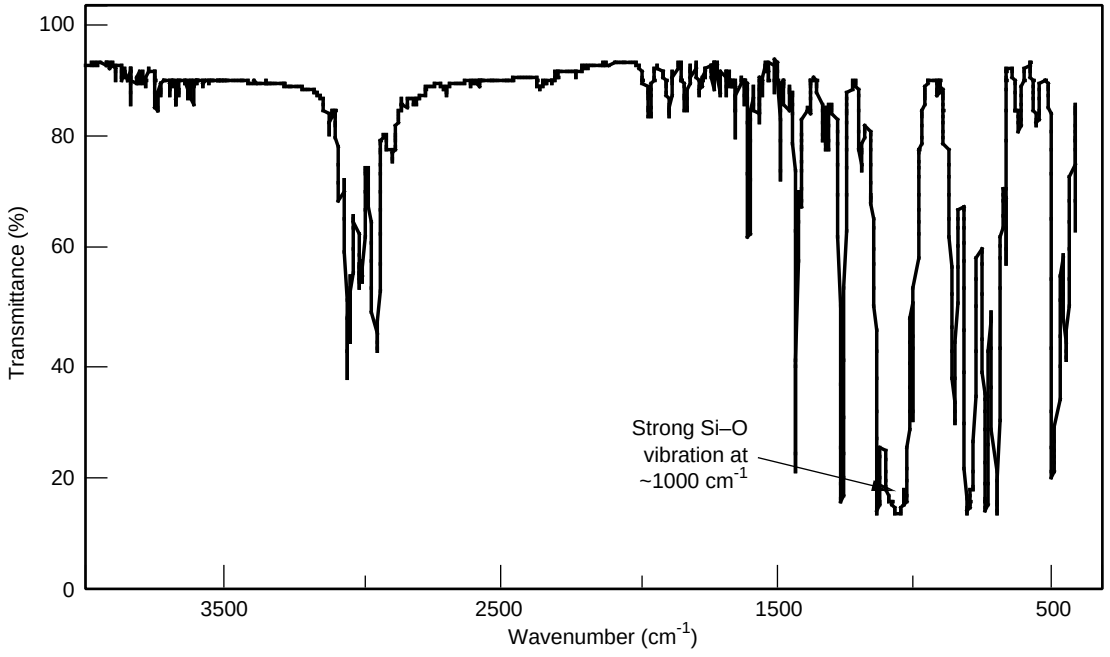
Attenuated total reflectance (ATR) is another technique for analyzing the surface of a solid (e.g., coatings) with little or no sample preparation. It can be used to analyze an opaque solid, semisolid, or liquid. It is a particularly good way to identify surface residues, and is applicable to adhesives, oils, greases or pastes, plastic films, paper and paper coatings, paints and finishes, fibers and fabrics, and foams and coated wires.

Diffuse Reflectance Spectroscopy

Diffuse reflectance spectroscopy is a good method for characterizing adsorbates on oxide or supported metal catalysts, and is also used for the identification of powders. It requires minimal sample preparation.

Gas Chromatography/FTIR

GC/FTIR can be used to identify the structure of organic mixtures. The gas chromatograph separates mixtures into their components. The components are introduced to the infrared detector as they elute, producing IR spectra for each compound. Submicrogram quantities of many volatile compounds in a mixture can be identified using GC/FTIR. Components of the mixture must be soluble in a suitable solvent and be volatile at temperatures below about 300°C (also see GC and GC-MS).



FTIR shows that used diffusion pump oil is silicon based and thus is not compatible with motor oil for disposal.